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# DISLOCATION MICROMECHANISMS & SCALE-FREE FLOW IN MICROCYRSTALS (PREPRINT)

**D.M. Dimiduk, C. Woodward, M.D. Uchic, S.I. Rao, E. Nadgorny, and P. Shade**

**Metals Branch**
**Metals, Ceramics and NDE Division**

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**14. ABSTRACT**
A frontier topic in computational materials science and mechanics is the development of a plasticity-modeling framework that naturally and accurately represents evolving length-scale effects and the consequences of the dislocation structure. Current studies show that important intrinsic "size effects" exist separately from an evolving excess dislocation density at mesoscopic scales. Understanding those effects forms an essential foundation for representing microstructural effects within predictive computational frameworks. Our studies focused on simulation, analysis and measurements of the plastic phenomena occurring in microcrystals having dimensions at the lower end of the mesoscopic domain, wherein the discrete and stochastic nature of the dislocation ensemble is visible. In prior work, we reported on selected experimental results for Ni crystals. The present studies examined the athermal flow response of micron-scale single crystals using large-scale discrete dislocation simulations (DDS) in 3d, under conditions closely related to our experimental methods.

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# Dislocation Micromechanisms & Scale-Free Flow in Microcrystals


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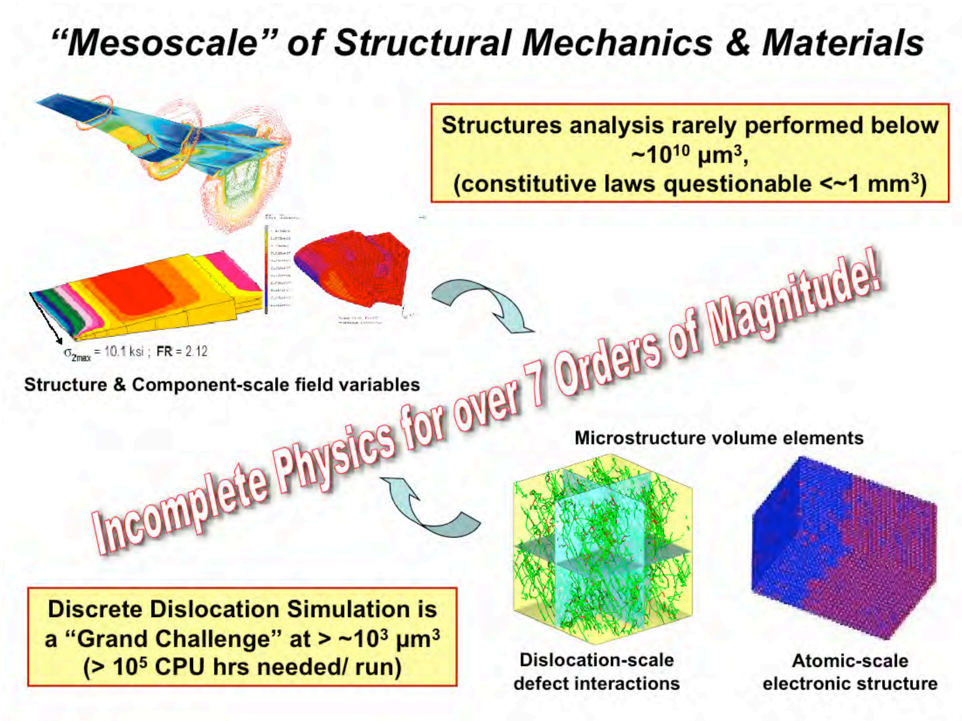
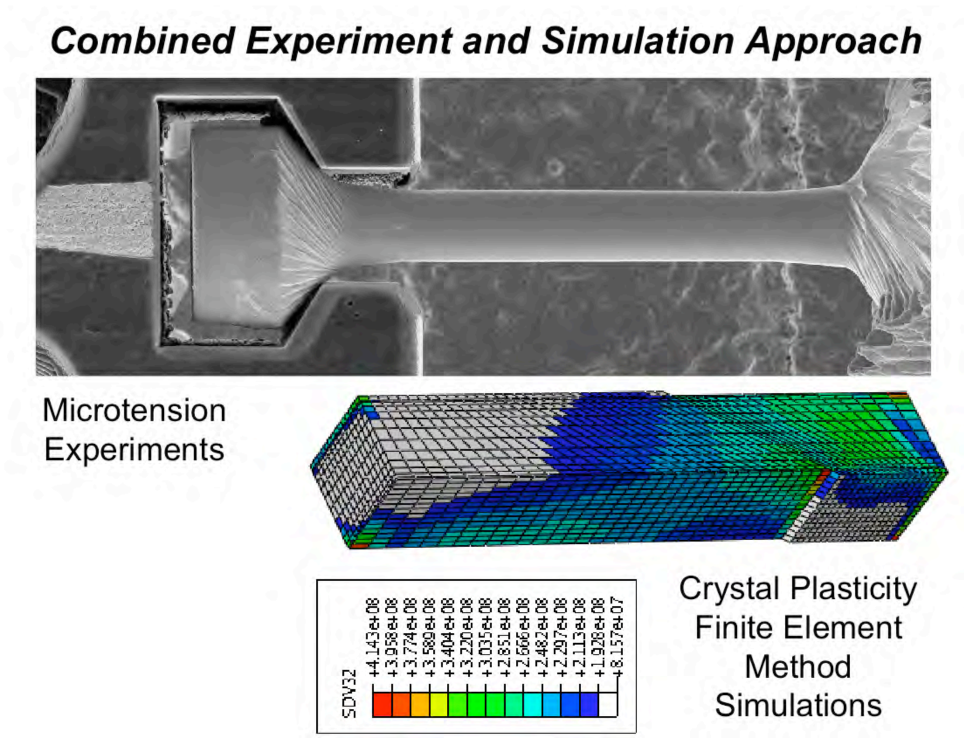
A frontier topic in computational materials science and mechanics is the development of a plasticity-modeling framework that naturally and accurately represents evolving length-scale effects and the consequences of the dislocation structure. Current studies show that important intrinsic "size effects" exist separately from an evolving excess dislocation density at mesoscopic scales. Understanding those effects forms an essential foundation for representing microstructural effects within predictive computational frameworks. Our studies focused on simulation, analysis and measurements of the plastic phenomena occurring in microcrystals having dimensions at the lower end of the mesoscopic domain, wherein the discrete and stochastic nature of the dislocation ensemble is visible. In prior work, we reported on selected experimental results for Ni crystals. The present studies examined the athermal flow response of micron-scale single crystals using large-scale discrete dislocation simulations (DDS) in 3d, under conditions closely related to our experimental methods. Uniaxial compression tests were simulated for cells ranging from 0.5-20 micrometers in edge length. Simulations were carried out for a range of initial dislocation densities, close to experimentally observed values. While the simulations revealed a clear cell-size dependence of the flow response, they also showed intermittency in the flow response over a finite range of strain and a stochastic nature to the observed flow stress. Similar results are known from experiments and were recently described within other reports of simulation studies. This presentation provides further descriptions and analysis of the intermittency and stochastic variation of flow response for micron-scale crystals, from both simulation and experiment, within the context of the arguments suggested in previous work.

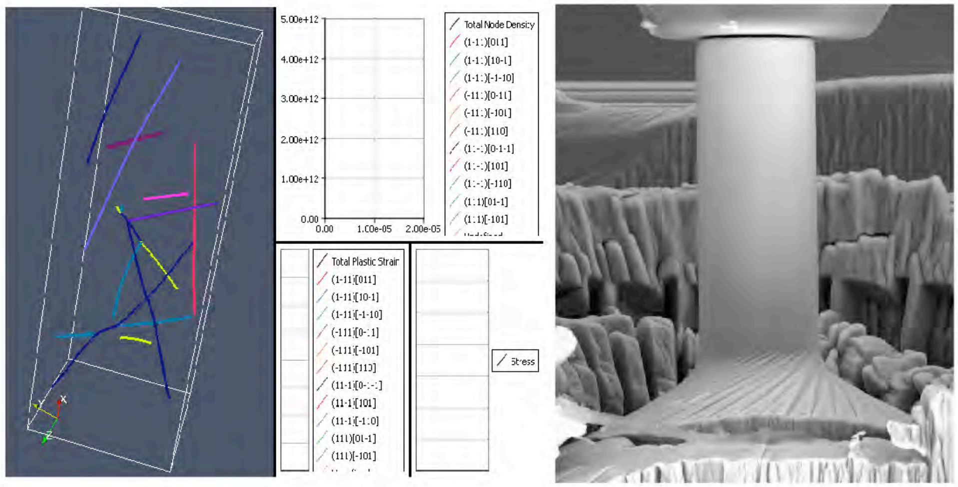



We approach the issue of microstructure-based design from the perspective of modeling capabilities. One must understand how the materials behavior mechanisms affect design and life management of parts. What we are doing today is working directly with the tools designers use and making them mechanistically better. We have constitutive modeling tools that work to about 1 mm3. At the near lattice dimensions, we also have good constitutive rules for dislocations. However, in between the physics is poorly understood and we only have phenomenology. The pacing research questions are centered around how to develop length-scale and microstructure-sensitive constitutive laws.




Our team has approached this challenge by working to develop combined microscale testing methods and simulation tools at the same scale. This slide shows a very interesting experimental result using our recently developed microtension testing method. While the details of these experiments will be presented in the next talk by Paul Shade, I just call attention to the localization deformation to slip planes, and note that propagation of slip after the initial strain burst takes place at lower applied stress. Our challenge is to represent that localization within a finite element simulation, and further, to understand the coupling of far-field loading to specimen geometry and internal stresses.

## *Combined Experiment and Simulation Approach*




Dislocation Simulations
*ParaDiS* code

Microcrystal Compression
Experiments

Similarly, we are have used 3d dislocation dynamics simulations to represent the behavior of microcompression experiments at essentially identical dimensional scales. The simulations have provided important new insights into the mechanisms governing the size effects on crystal strength. Now, we are trying to use the method to understand dislocation ensemble effects on strain avalanches.

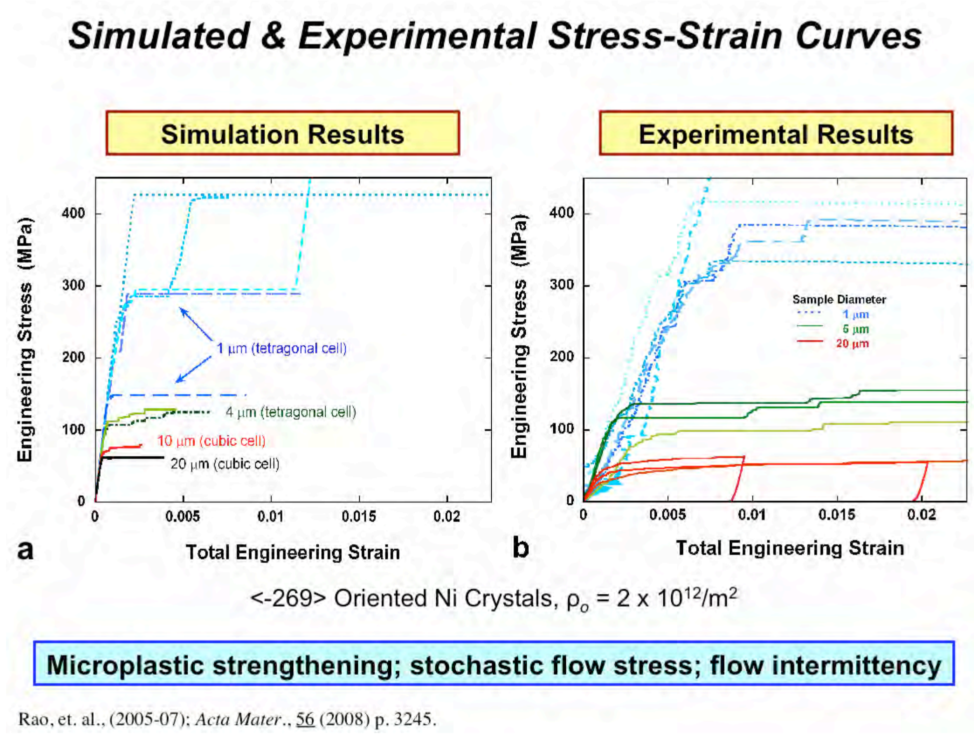
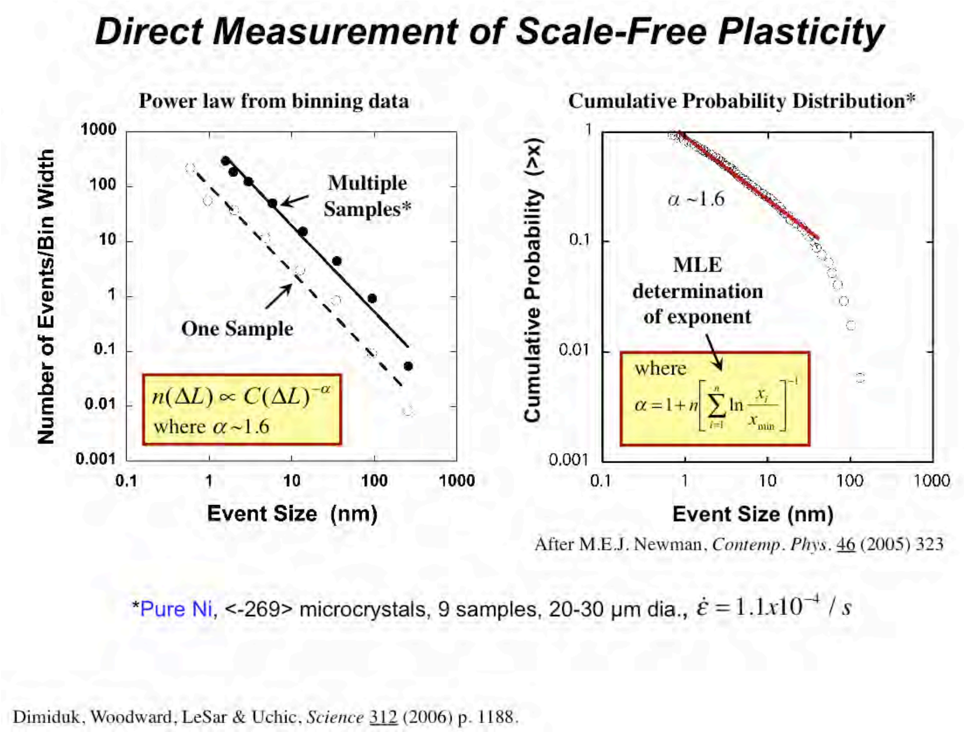



Chart shows the simulation results for stress versus strain in comparison to the stress-strain results from experiment. The results are plotted on the same scale. Note that the qualitative agreement is excellent and the principal phenomenology of the experiments is captured in the simulations. The samples exhibit a hardening relative to bulk materials at near zero strain, followed by a small strain interval of intermittent flow and rapid strain hardening at a high. For the experiments, initial analysis showed that the strain bursts or avalanches exhibit scale-free power law probabilities of avalanche size. The remainder of the talk reviews that analysis and discusses issues both with the analysis and the interpretation of avalanche events.




This slide shows the culmination of the experimental measurements described previously, where the number of random events has been plotted as a function of the size of the events in the figure on the left. The open circles correspond to measurements from a single sample, while the filled circles correspond to an aggregate from multiple samples (upwards of 10 samples). the number of events versus event size follows a power law form over 3 orders of magnitude—scale free flow—with the value of the exponent equal to 1.6. This value can be related to the energy dissipated by dislocation glide through the crystal, and realistically this value of energy dissipation is simply related to the number of dislocations that participate in these random avalanches (i.e., the avalanche magnitudes vary over 3 orders of magnitude, therefore the number of dislocations that participate or dislocation density also varies by this amount).

## *Selected Dislocation Avalanche Studies*

- Pond, (1971) Slip band displacements versus time
- Neuhauser (1983) Extensive study & review of slip band evolution
- Miguel, Weiss, et al. (2001) Acoustic emission and 2d dislocation dynamics
  - Probability of dislocation velocities in periodic cell
  - Long range interactions between straight parallel dislocations
  - Pinning via dipole walls
- Koslowski, et al. (2004) Phase field simulations of dislocations moving through point-obstacle (forest dislocation) arrays
  - Long range interactions (Orowan looping)
- Dimiduk, et al. (2006) Microcrystal direct measurements of probability of total axial displacement (area swept or strain)
- Zaiser, Csikor, et al. (2007) Theory and simulation assessment of largest avalanches
- Weiss, et al. (2008) Experiment and theory for largest avalanches
- Devincre, et al. (2008) 3d dislocation dynamics of forest cutting in a periodic cell show power law scaling of strain increments
  - short range forest processes control capture & release
- Fressengeas, et al. (2008 preprint) Macrocrystal experiments & field theory simulations
  - Maximum avalanche strain rate (group velocity of dislocations) is fundamental
  - Long range forces, short range event control

Self explanatory; point out potential inconsistencies regarding long-range versus short-range interaction control

## *Selected Issues Regarding Dislocation Avalanches*

- Largest avalanches expected to contribute largest part of strain in any experiment; but not seen for large specimens—why not?

- What is and what controls the avalanche cut-off (correlation length)?
  - Zaiser, et al, (2007), Theory: $X_{max}$ proportional to Sample 'Size' (L), Machine Stiffness (M) & Hardening Rate (H)
  - Weiss, et al, (2008) Experiment & Theory: $X_{max}$ proportional to L & M; H does not matter

- Has the appropriate experiment been done?
  - For pole sources few limits on $X_{max}$
  - For creep loading M is excluded (constant far-field driving force)
  - For low dislocation density, H tends to zero

  … $X_{max}$ in small samples may only be limited by boundary conditions of testing

- Is there a relationship between avalanche size and the correlation length of the dislocation forest (perhaps related to mean free path?)

- Effects of other details
  - Strain rate and deformation Stages (0, I, II & III)
  - Cross-slip and strain hardening mechanisms
  - Reconciling simulation and experimental data

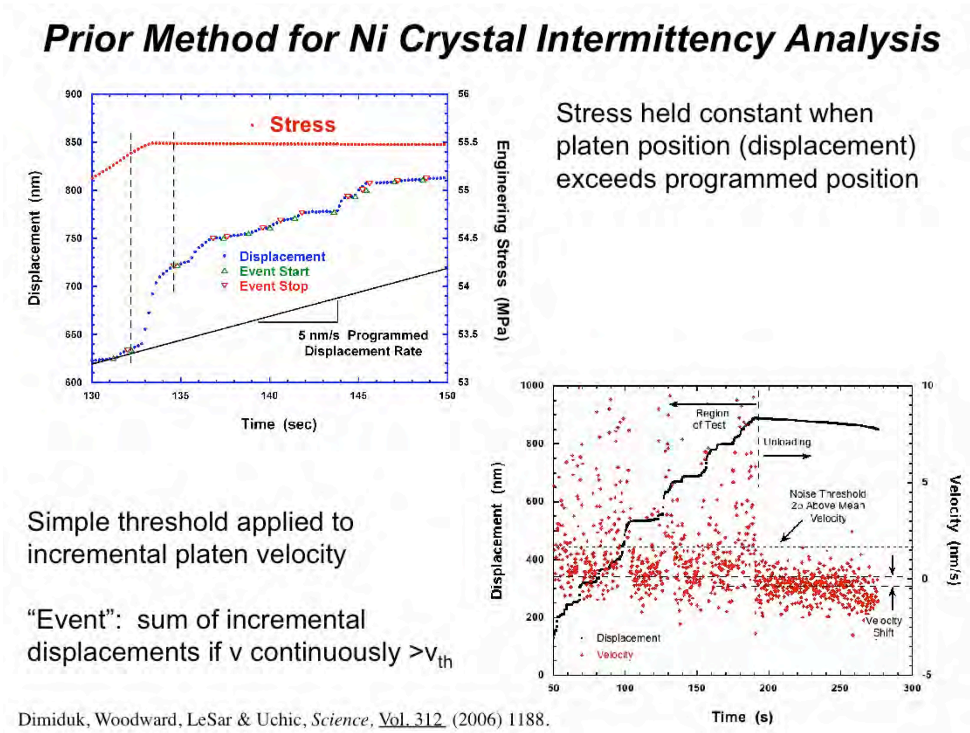
- Demands rigor in analysis methods to handle diverse nature of observed responses

Self explanatory

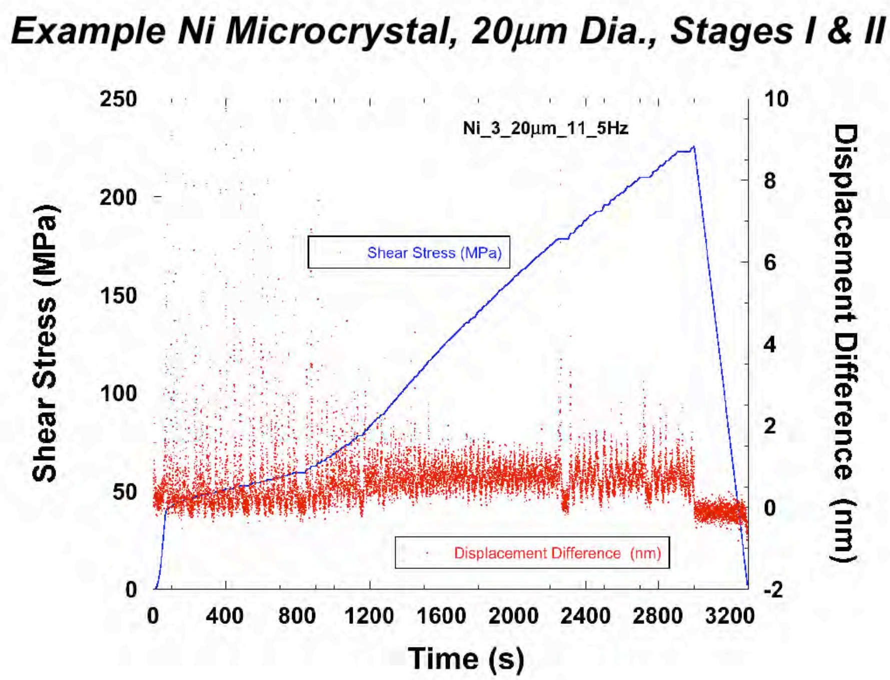
## *Unsolved Issues With Microcrystal Experimental Data*

- Broad variations of “mean velocity”
  - Platen velocity variations inconsistent with constant threshold method
- Local variations in “mean velocity”
  - Platen motion or loading mode changes; during avalanche events
- Data collection frequency effects
- Effect of “Continuous Stiffness Measurement” signal
- “Goodness-of-fit” for power laws & parameters
- Automation of analysis tools

Self explanatory




After the initial experiments, we suspected a regime of scale-free power law avalanches, so we made a statistical analysis of the experimental data. Examination of the displacement record showed clear intervals for which the platen velocity exceeded the programmed rate during periods of constant stress. The velocity signal was examined using a simple threshold technique.




By way of example, the plot shown of the stress-strain and incremental platen displacement behavior of a 20 micron Ni sample, reveals why a simple threshold method will not work very well. The displacement difference signal experiences a pronounced shift in background level, both upon change in the overall deformation stage, and more locally when large avalanches occur. Thus, a new method is needed that more accurately follows the background response.

## *Introducing New Methods for Analyzing Data*

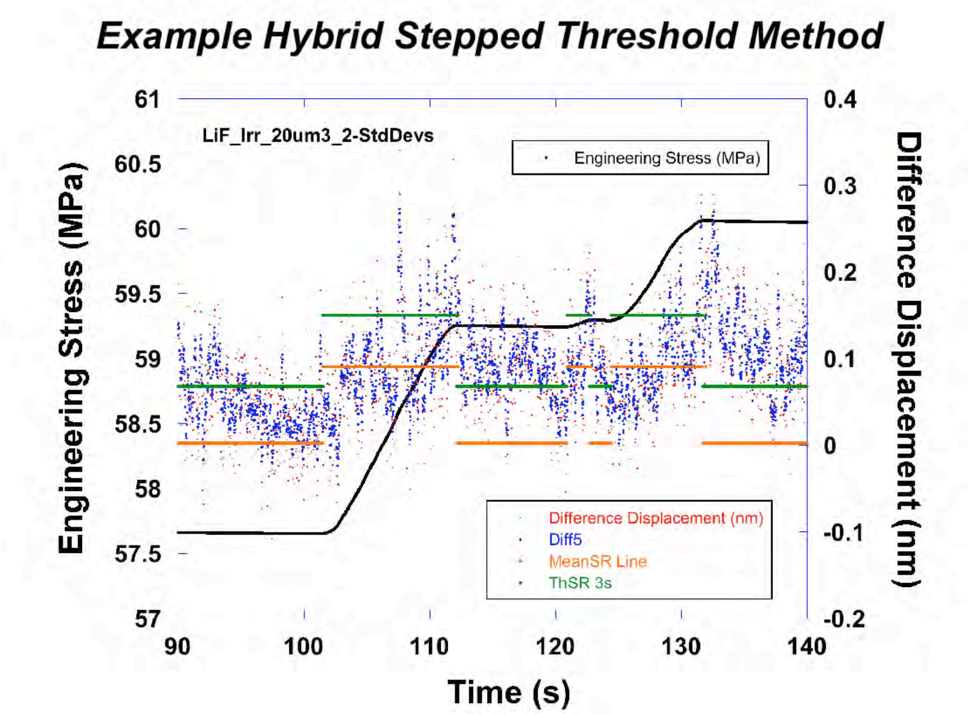
- Three key advances
  - Hybrid, stepped threshold to match loading mode
  - Window averaging (# of time records)
  - Noise threshold (mean + *n*-sigma)

- Shows variations in $X_{min}$, $X_{max}$, $N_{total}$, Alpha, & "goodness-of-fit" to power laws

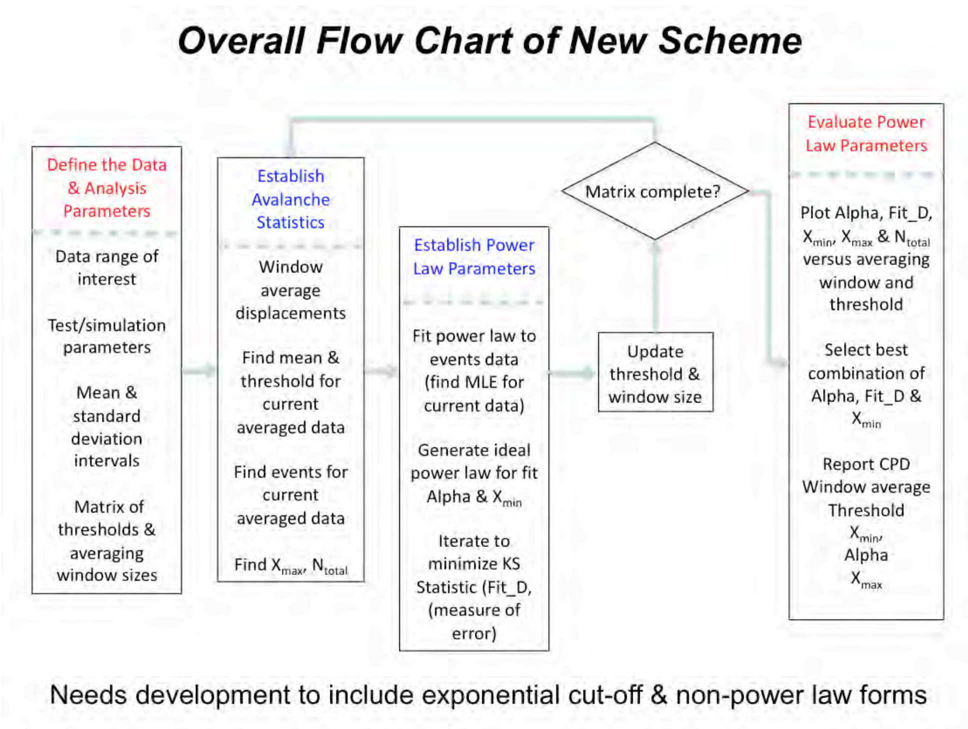
- Methods Based on Key Paper:
  - Clauset, Shalizi & Newman, submitted to SIAM Reviews (2007); Preprint at arxiv:0706.1062.
  - http://www.santafe.edu/~aaronc/powerlaws/

- Applicable to both experimental and simulation data

Recently, we developed a method that adjusts the threshold to the loading mode for our hybrid loading method. Further, in the new method we enabled a technique for window averaging the displacement records over several time intervals to enhance the signal to noise ratio. Finally, the new method adjust that windowing variable over some specified range, and then processes the data over a range of threshold levels for each window. From the new technique, one can examine variations in the minimum and maximum events detected, track the variation in the number of avalanches detected for those variations and, finally, detect the variation in the power laws exponent (alpha) and the quality of the power law fit to the data using the KS statistic. As for the quality of the power law fit and our new method for analysis, much of it is build on the descriptions that you may find in the review paper by Clauset et al, as noted on the chart. Our analysis method has adapted the Matab routines posted on their web site (also shown).

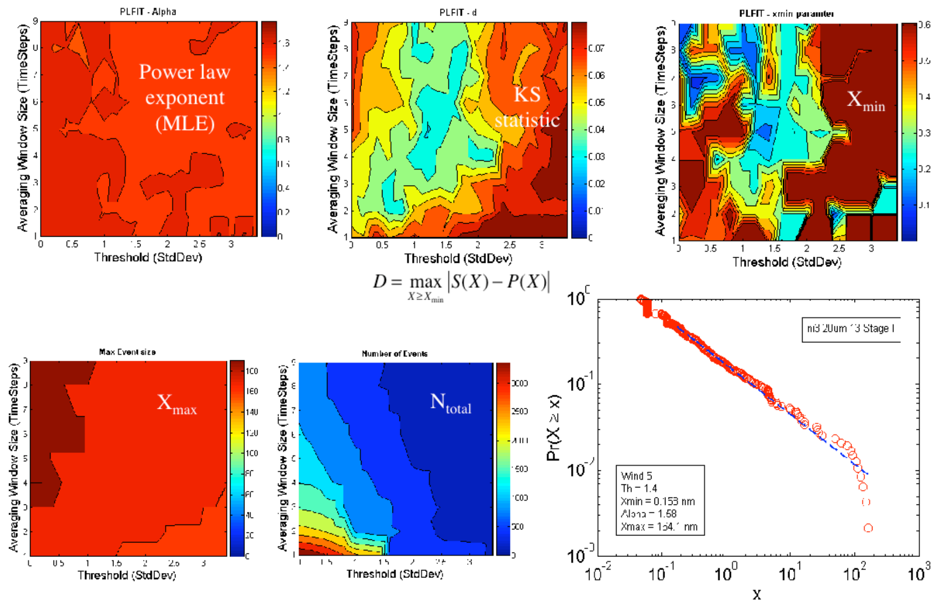



This chart shows and example the stepped threshold method relative to a enlarged view of the displacement signal for a LiF sample. Note that the new method follows the so-called avalanche aftershocks that would be simple eliminated by a simple threshold method. Each break in the threshold green line represents a change in the programmed loading mode from a creep to a loading interval, or vie versa.




The chart shows a overall flow chart schematic of our new procedure for data analysis. The same procedure is used for both experimental and simulation results, with one exception. For the simulation results, the displacement record versus time is not generated on a regular grid since it is a result form the ParaDiS code that automatically adjusts the time stepping with the dislocation reactions. Thus, for the simulations, there is a fitting and re-sampling procedure used to regularize the time stepping before the remainder of the analysis proceeds.

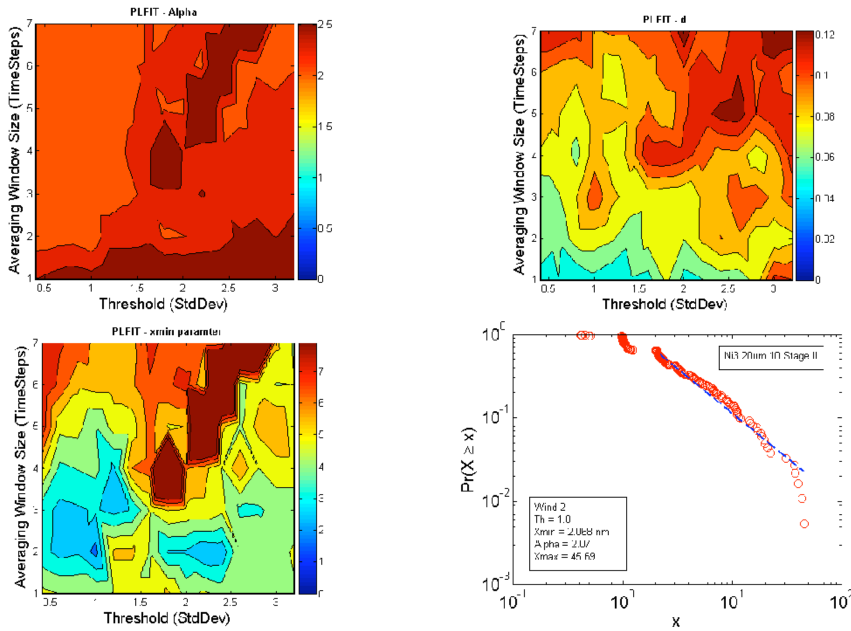
## *Example: Pure Ni, 20µm, Stage I Flow*




New method confirms previous exponent, *α* ~1.6, at $10^{-4}$/s; much better statistics

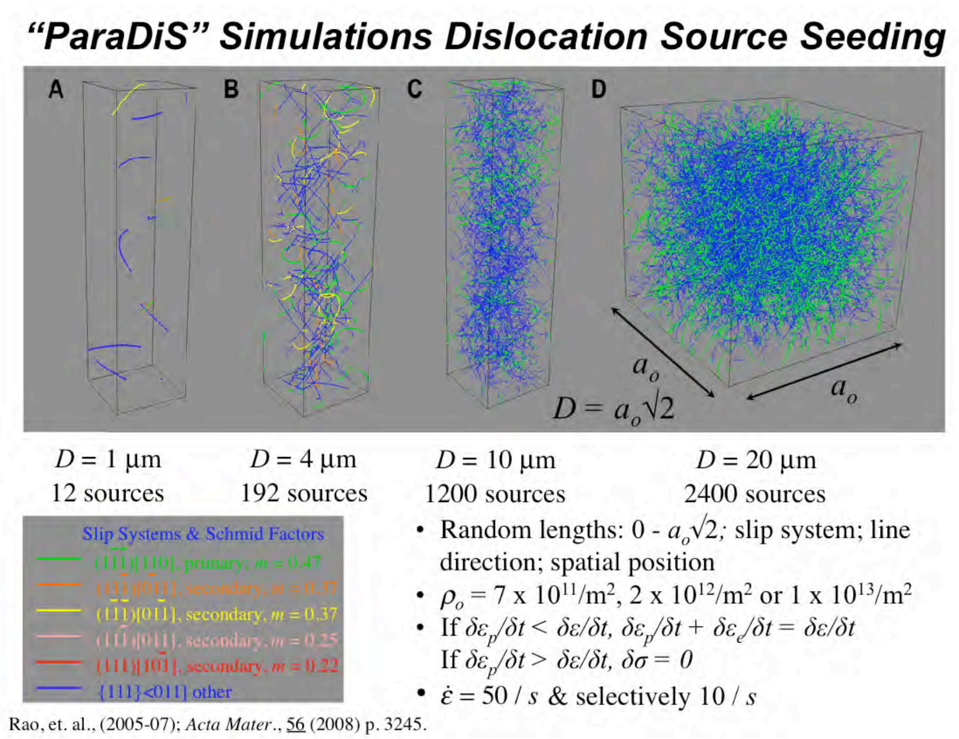
This chart shows an example for 20 micron Ni microcrystals of the types of results we obtain from the new analysis method and codes. From upper left to lower right the figures show contour plots of the avalanche displacement size, the KS statistic measure of the goodness of fit to power law behavior, the value of the minimum avalanche detected that is consisient with power laws behavior, the maximum detected avalanche size and the total number of avalanches detected. All for the contour plots are shown for data averaging window size versus threshold level coordinates. The threshold level is expressed in units of standard deviation. The final plot on the lower right is the cumulative probability of displacement event sizes for an averaging window of 5 and a threshold of 1.4. For these conditions, the scaling exponent found via the continuous linear threshold method is confirmed, but there are now much more events detected and better statistics.

## ***Power Law Results: Pure Ni, 20µm, Stage II Flow***



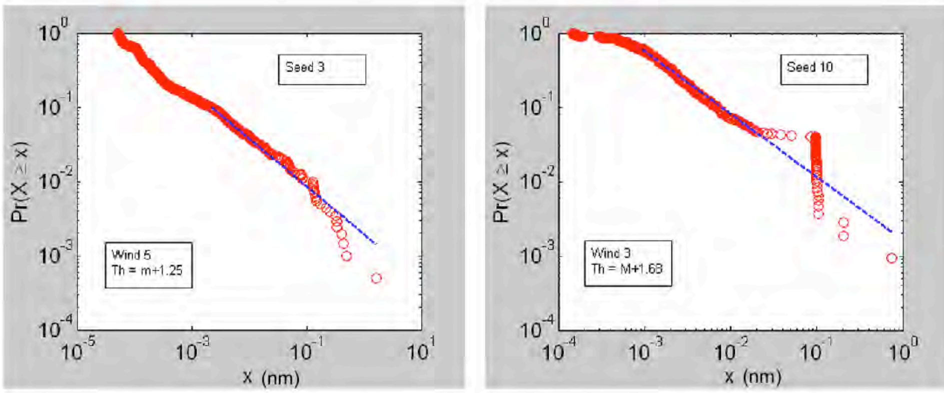

Exponent increases, $\alpha$ ~2.1; $X_{max}$ decreases by ~10x compared to Stage I

Using the new technique to examine the stage II flow regime, one can see that the scaling exponent changes. The Value for stage I is 1.6 while that for stage II is ~2.1. This means that as the substructure is refined, the range of avalanche size decreases. Also, the maximum detected event decreases by about 10x.




Last part of this presentation begins to examines the avalanche behavior observed for discrete dislocation simulations. As series of simulations for the cell sizes shown, were conducted using the ParaDiS code. Each simulation had a random selection of Frank-Read sources. For this series of simulations, cross-slip was not permitted, so the results reflect purely athermal behavior. One point to note is that the free surfaces permit formation of single-armed sources right from the onset of plasticity. These dominate the behavior of the smallest cells.

## *Power Law Results for 1 μm Cell Simulations*

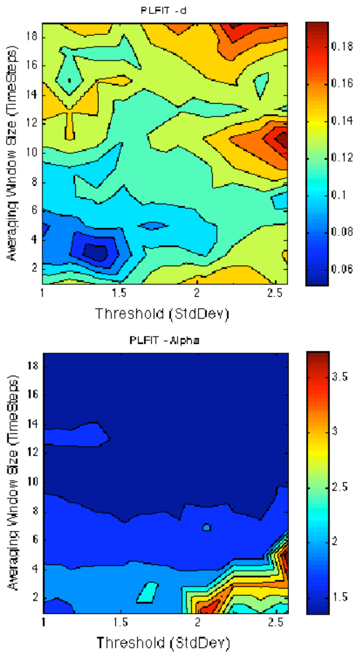


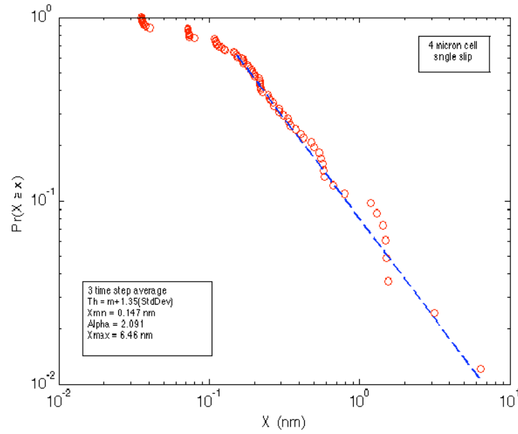

| Multiple active sources | Dominated by single source |
|---|---|
| $\alpha = 1.65$ | $\alpha = 1.81$ (perhaps) |
| 'experiment–like' | 'other' |

Simulations comprise two groups of results: 'experiment like' & 'other'

Analysis of the simulation cells is still underway. However, for the one micron cells, the 10 initial seeds appear to separate into two populations. On the left the cumulative probability distribution of avalanche sizes shows an experiment-like response. On the right, a completely different behavior emerges. For this population of simulations, there is a sharp peak in the distribution at about 0.1 nm. The avalanche size corresponds to single dislocations sweeping the glide plane, or single-armed sources. It will interesting to see how these distributions are affected by cross-slip and larger simulation sizes.

## *Power Law Results for a 4μm Cell, Single Slip*



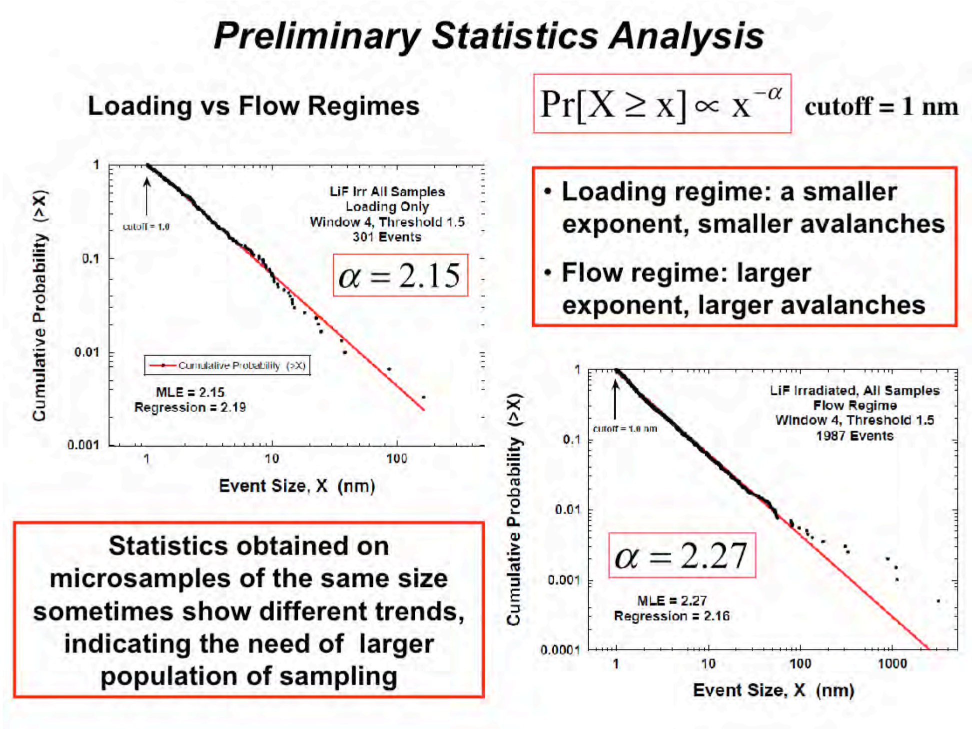




Multiple active sources

$\alpha = 2.09$

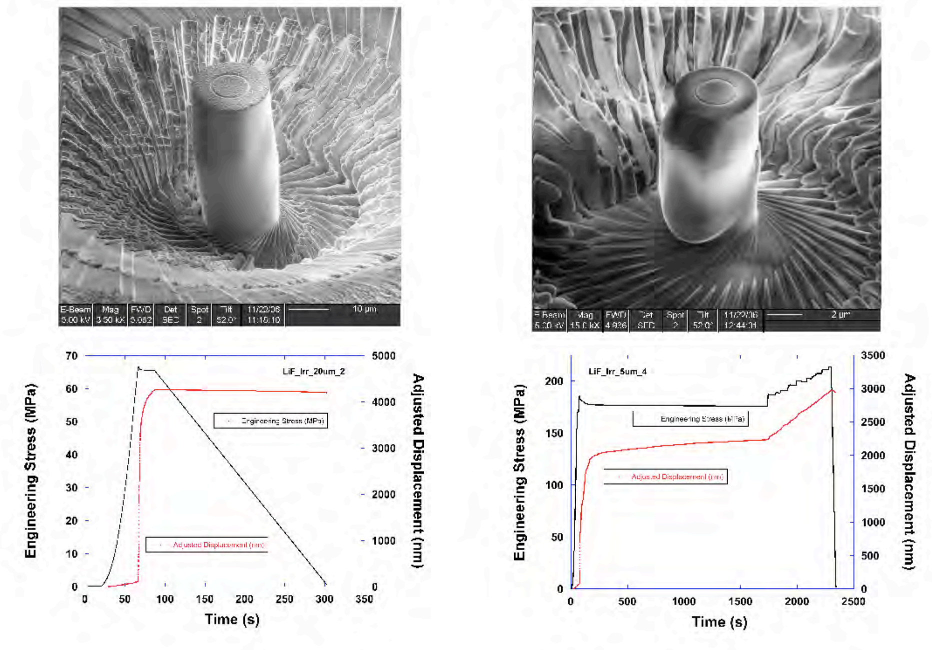
'experiment–like'

An examination of a single simulation at the 4 micron cell size reveals an experimental-like cumulative probability of avalanches. Again, the results tend to indicate that dislocation interactions control the range of the avalanche size. What is still unresolved is why these simulations show a power law exponent that is stage II – like, while the flow curves (strain hardening rate) for the simulation is stage I - like.



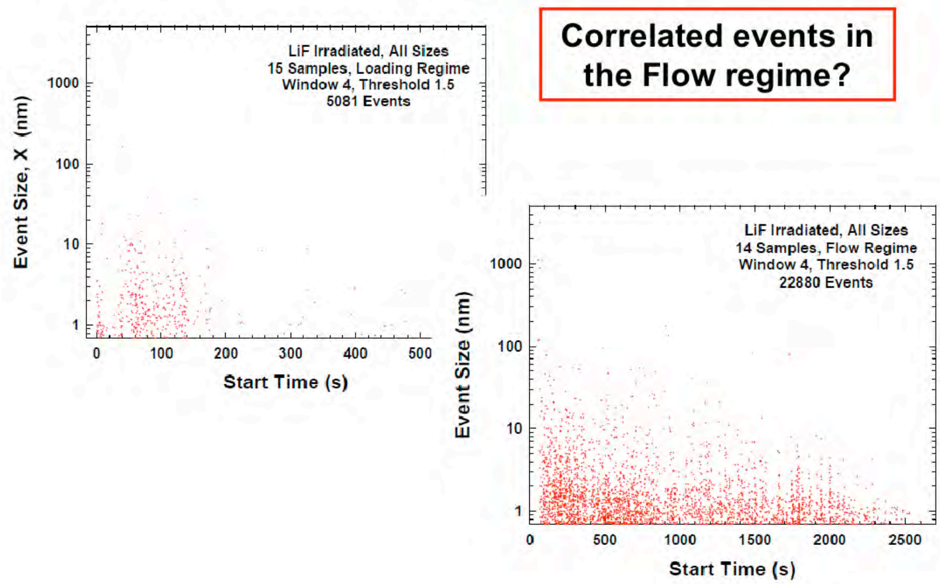

For the final few slides, we show some of the aggregate results for LiF crystals. This chart shows a comparison between the loading only regime (elastic to plastic transition) and the flow only regime (linear stage I hardening). Both are for irradiated LiF that contains point defects. As aggregates, the sample show a larger scaling exponent for the flow regime, somewhat counter to expectations, but the flow regime also shows extremely large avalanches that do not follow power law behavior. We have called these nucleation controlled events. Importantly, if attempts the same comparison for individual sample data, the results are mixed, suggesting that one needs good statistics ( a large enough event population).

## *Irradiated LiF, Largest Avalanches Occur at Start*

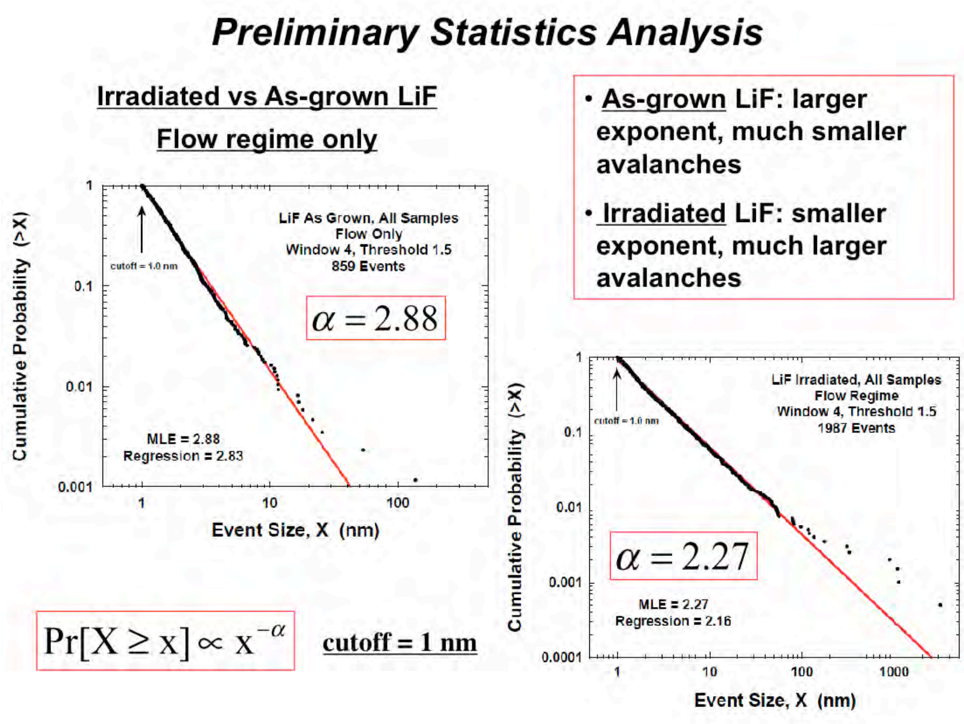



This chart shows two examples of very large avalanche events for 20 and 5 micron LiF samples. At the lower part of the chart, one can see the stress-time and displacement time records for the tests. Note from the red lines that a very large displacement interval takes place near the beginning of the test. Examining the SEM images at the top of the chart, one can observe that the zones of very fine slip have appears on the sample. For the 5 micron sample on the right, two slip bands formed and appear to have evolved independently of each other. Since these crystals had an initial dislocation density of less than 10^9/m^2, we envisage that pole sources formed in the crystal and that these looped around the sample cross-slipping at the surface and forming fine continuous slip zones. All of this occurred under conditions of high stress since those sources had to be nucleated, thus the system was in an over driven state during the avalanche period, but yet had little opportunity for strain hardening.

## *Preliminary Details: Avalanche Size vs Start Time*




Another way to examine the nucleation controlled events is to plot the avalanche event size as a function of its start time during the testing. These two plots show such data for the aggregate of samples of irradiated LiF. Note that none of the large avalanches occur during loading. On the other hand, the largest events always take place very near the start of the flow regime. Our thinking is that the flow regime is defined by readily multiplying dislocations. Thus, the events take place when stress are large enough to multiply dislocations, but before there is a high dislocation density to trap dislocations and to keep their sizes small.




Finally, this chart compares the Irradiated LiF with As Grown material, for the flow regime only (linear stage I hardening). The data show that avalanches are of smaller sizes in the as grown material, even though the initial dislocation densities of the two materials are essentially equivalent. It is likely that the ~10x lower friction stress for the as grown material permits more dislocation-dislocation interactions to take place at lower stresses than for the irradiated material. Also, when dislocations are released at the start of an avalanche, the driving force is greater during motion because of the higher stress in the irradiated material.

## *Concluding Comments on Selected Issues*

- Largest avalanches…not seen in large specimens—why not?
  - *Large avalanches are seen in microcrystal experiments (>4.3 μm)*
  - *Appear to limited by driving force and strain hardening*
  - *Other experiments may be tailored to show even larger avalanches*
- What is and what controls the avalanche cut-off (correlation length)?
  - *Sample size and machine stiffness are relevant factors, but do not dominate our microcrystal experiments*
  - *Strain hardening is the most important fundamental effect, influenced by cross-slip*
- Low Dislocation density and likely pole sources permit very large $X_{max}$ for microcrystals
- Is there a relationship between avalanche size and the correlation length of the dislocation forest (perhaps related to mean free path)?
  - Should be, just as SHR is related to mean free path
  - Needs further investigation
- Effects of other details
  - Strain rate and deformation Stage 0, I, II & III effects
  - Effects of cross-slip and strain hardening
  - Reconciling simulation and experimental data

Self explanatory. We summarize by commenting on a few of the questions raised at the beginning of the talk.